

EUROPEAN PATENT OFFICE

Patent Abstracts of Japan

PUBLICATION NUMBER : 05098301
PUBLICATION DATE : 20-04-93

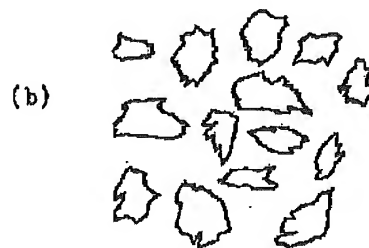
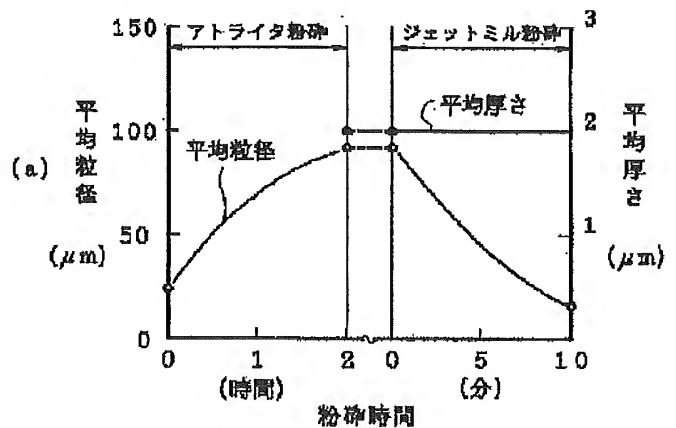
APPLICATION DATE : 07-10-91
APPLICATION NUMBER : 03259065

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INT.CL. : B22F 1/00 B22F 9/04 G12B 17/02

TITLE : FLAT FINE METAL POWDER AND ITS PRODUCTION



ABSTRACT : PURPOSE: To produce a flat fine metal powder having ductility and with the average particle diameter and the average thickness specified.

CONSTITUTION: A metal powder having ductility is obtained by the atomization method, the powder is wet-crushed by an attritor to $\leq 2\mu\text{m}$ thickness and flattened, and the flat powder is crushed by a jet mill to the fine particles having $\leq 20\mu\text{m}$ average diameter. At this time, efficiency is increased by the combination of the crushing by the jet mill and a flash classifier. The flat fine metal powder obtained by this invention is angular, and the powder is effectively applied to the soft magnetic powder to be used in a magnetic shielding paint.

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(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平5-98301

(43) 公開日 平成5年(1993)4月20日

(51) Int.Cl. ⁵	識別記号	庁内整理番号	F I	技術表示箇所
B 2 2 F 1/00	B	7803-4K		
9/04	E	9157-4K		
G 1 2 B 17/02		6843-2F		

審査請求 未請求 請求項の数4(全6頁)

(21) 出願番号 特願平3-259065

(22) 出願日 平成3年(1991)10月7日

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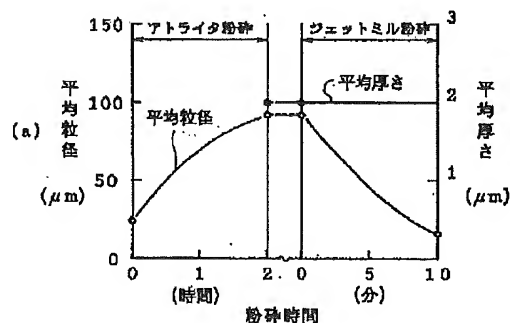
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(54) 【発明の名称】 扁平状金属微粉末およびその製造方法

(57) 【要約】

【目的】 延性を有し平均粒径が $20\mu\text{m}$ 以下、平均厚さが $2\mu\text{m}$ 以下の扁平状金属微粉末およびその製造方法を提供する。

【構成】 延性を有する金属粉末をアトマイズ法で得た後、この金属粉末をアトライターで湿式粉碎することで厚さ $2\mu\text{m}$ 以下まで扁平状に加工し、この扁平状粉末を乾燥後、ジェットミルで粉碎して平均粒径 $20\mu\text{m}$ 以下に微粒化する。この時、ジェットミルでの粉碎と気流分級装置を組み合わせたシステムとすると、効率が上がる。本発明方法で得られる扁平状金属微粉末は角ばった形状をしており、磁気シールド用塗料に使用する軟磁性粉末に適用すると、特に有効である。



のに最適な形状と大きさを有する扁平状金属微粉末およびその製造方法を提供しようとするものである。

【0007】

【課題を解決するための手段】本発明者は種々検討を重ねた結果、磁気シールド用塗料に用いる軟磁性の扁平状金属粉末の形状は、気流粉碎機で粉碎したものであって、微粉末のふちが角ばっており、かつ、平均粒径が20 μ m以下、平均厚さ2 μ m以下であるのが最適であることを見出し、さらにこのような形状で粒径や平均厚さの揃った微粉末を得るためには、アトライターで湿式粉碎した後、乾燥し、次に気流粉碎機で粉碎する2段階の粉碎方法が効率が高いことも見出して本発明を完成したものである。

【0008】具体的には、本発明の金属粉末は延性を有する金属粉末であって、気流粉碎によって微粉化され、平均粒径が20 μ m以下、平均厚さが2 μ m以下であり、角ばった形態をしたことに特徴がある扁平状金属微粉末であり、そして、組成的にはパーマロイ、センダスト、またはパーメンダ等に代表される軟磁性金属粉末であることの特徴とするものである。さらに本発明の扁平状金属微粉末を得る方法は、アトライターに代表される媒体攪拌式粉碎機により湿式粉碎して乾燥した後、ジェットミル等のような気流粉碎機により粉碎して微粉化することの特徴とするものであり、気流粉碎に続けて気流分級で分級して、寸法的に安定な扁平状金属微粉末を得ることを特徴とするものである。

【0009】

【作用】延性を有する金属粉末を扁平状に加工する方法としては、ロールにより圧延する方法やボールミル、アトライターなどの粉碎機で粉碎する方法が知られている。しかし、ロールにより圧延する方法では、本発明で目標とする厚さ2 μ m以下の扁平状に加工することは非常に困難であり、粉碎機で粉碎した場合には、目標とする平均粒径20 μ m以下、平均厚さ2 μ m以下の粉末を得るには長時間の粉碎が必要である。粉碎機の中でも粉碎エネルギーの最も大きい媒体攪拌式粉碎機であるアトライターは、比較的短時間で微細な粉末を得ることができるが、それでも工業的に利用できる平均粒径20 μ m程度のFe-Ni系合金粉末を粉碎した場合、目標とする粒径、形状の粉末を得るには10時間以上の粉碎時間が必要である。また、アトライターによる粉碎では、金属粒子同士が粉碎中に凝集、凝着するのを防止するため、アルコール類などの溶液中に分散させた状態で湿式粉碎する必要がある。

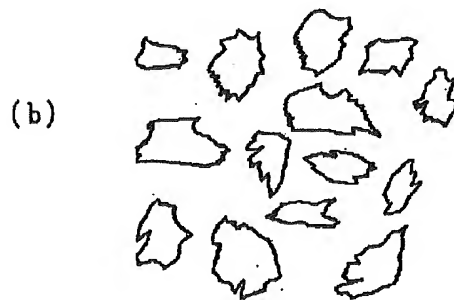
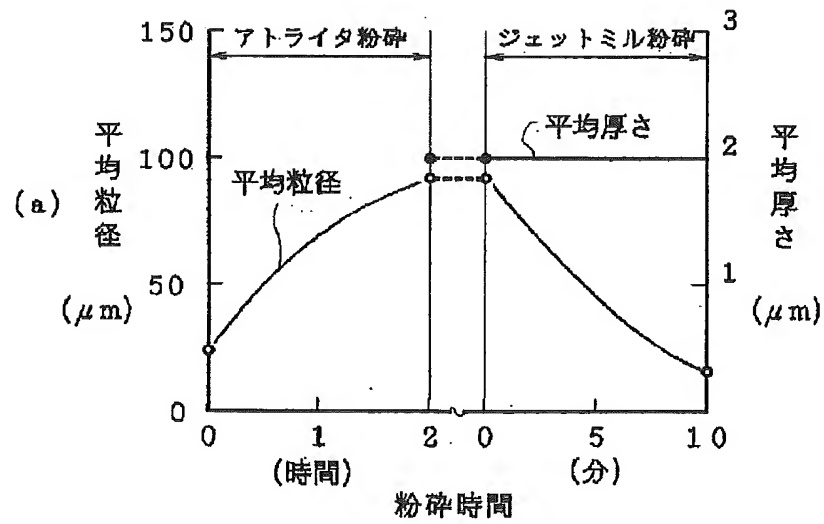
【0010】本発明者は、延性を有する金属粉末のアトライターによる湿式粉碎の過程を観察した結果、金属粒子は、粉碎過程の初期段階においてすでに目的とする厚さの扁平状粒子に加工されていること、しかし個々の扁平状粒子のかどは丸味をおびているので、これらの扁平状粒子が分断されて微粒化するのに非常に長時間の粉

を必要とすること、および扁平状粉末同士は十分に分散された状態でないと容易に凝着して、扁平度が低下することを発見した。すなわち、延性を有する金属粉末に対するアトライターでの湿式粉碎は、金属粒子を平均厚さ2 μ m以下に短時間で加工する作用に対しては非常に有効ではあるが、一方で扁平状金属粒子を分断し微粉化する作用に対しては効率が悪いということがわかった。この原因は、アトライターでの湿式粉碎は、基本的には粉碎機内の鋼球同士の衝突により、金属粒子が衝撃作用を受けて粉碎されるが、この衝撃作用は、金属粒子の扁平化には有効であるが、扁平粒子の微粉化に対しては効率が悪いと、機械的に湿式粉碎した延性の高い金属粒子はかどが丸味をおびており、さらに加えられる粉碎力でもなかなか粉碎され難いためと考えられる。

【0011】ところで、延性を有する金属粉末の粉碎過程における材料の機械的特性の変化を考察してみると、粒子が扁平状に塑性変形する過程において、延性を有していた粒子も、粒子が扁平状に変形した後は、材料が加工硬化して脆化していることが容易に推測される。そこで種々検討した結果効率よく扁平状微粉末を製造するためには、粒子を2 μ m以下の扁平状にする第1段階の粉碎をアトライターで短時間でを行い、第2段階の粉碎は、脆化した扁平状粒子を微粉化する作用が強い粉碎方法で行うことが有効であることが判明した。さらに第2段階で行う脆化した扁平粒子を微粉化する作用が強い粉碎方法としては、ジェットミルなどによる気流粉碎法が有効であることを見出した。本発明の方法による気流粉碎によれば、従来とは全く異なる形態のかどが角ばった粉末が得られる。本発明でいう気流粉碎法とは、高速の気流中で、粒子間の衝突や粒子と障壁との衝突により、粒子を粉碎するものである。本発明で対象とする脆化した扁平状粒子は、その形状の効果により、高速気流中で高い運動エネルギーを受け、なおかつ材料が脆化しているため気流粉碎機を用いれば分散状態を維持した状態で粉碎が終了するまでその形状がふちが角ばった状態を維持できるので容易に粒子が破壊されて微粉化するのである。

【0012】さらに微粉末の分級方法としては、一般に気流分級法が適用されているが、この気流分級法を、気流粉碎法と組み合わせて連続的に行い、気流粉碎した粉末を、その気流によって気流分級し、気流分級されて粗い粒子として選別された粉末を再び、気流粉碎機に戻して粉碎し、微粉のみを回収するように装置と気流の配管を組むと、連続的に100%の歩留りで、扁平状金属微粉末を製造でき効率的である。本発明の扁平状金属微粉末は、装飾用、防錆用塗料用としても使えるが特に電磁気的な特性を利用したシールド用塗料として使用すると最適である。平均粒径が20 μ mを越えると塗布膜の表面粗度が粗くなり、そのため電磁気特性が不均一になりノイズ発生の原因となる。また平均厚さが2 μ mを越えるとシールド特性が悪くなるので、平均粒径20 μ m以

【図1】

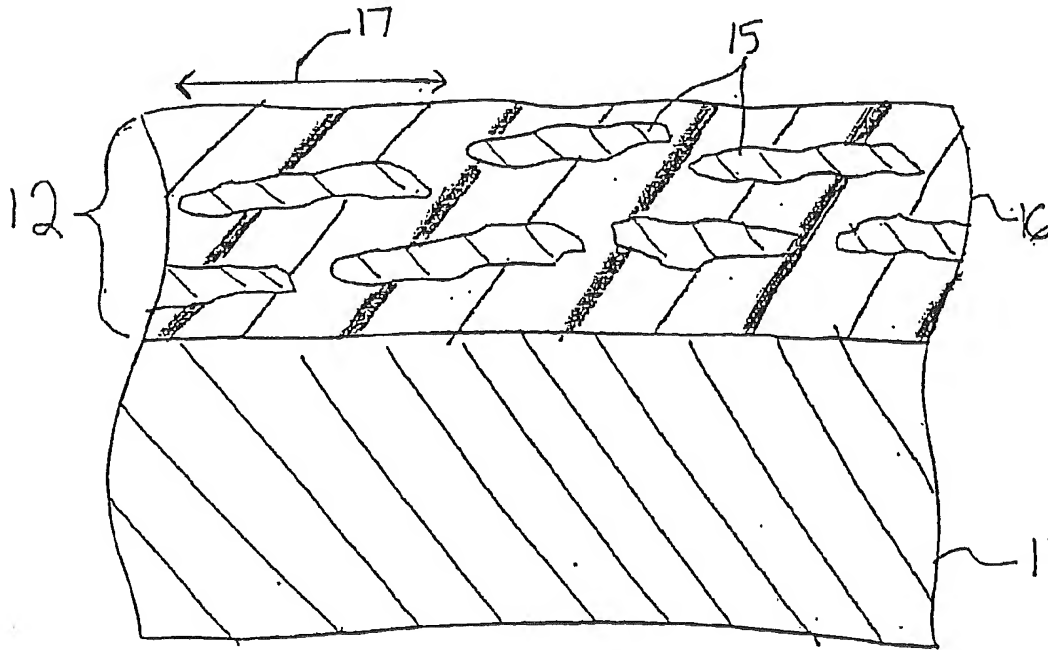




US 20030008131A1

(19) **United States**(12) **Patent Application Publication**
Paris et al.(10) **Pub. No.: US 2003/0008131 A1**(43) **Pub. Date: Jan. 9, 2003**(54) **METHOD FOR MAKING RADIATION
ABSORBING MATERIAL (RAM) AND
DEVICES INCLUDING SAME**(75) **Inventors: Henry G. Paris, Chattanooga, TN
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nooga, TN 37401**(21) **Appl. No.: 10/189,654**(22) **Filed: Jul. 3, 2002****Related U.S. Application Data**(60) **Provisional application No. 60/302,768, filed on Jul.
3, 2001.****Publication Classification**(51) **Int. Cl.⁷ G21F 1/10; B32B 5/16; G21K 1/10;
H01C 1/00; H01B 1/02**
(52) **U.S. Cl. 428/328; 428/331; 523/137;
252/513**(57) **ABSTRACT**

A method for making a radiation absorbing material (RAM) coating may include providing an iron-silicon alloy powder, forming the iron-silicon alloy powder into flakes, and passivating the flakes. The method may further include selecting passivated flakes having a desired size, and combining the selected passivated flakes with a carrier to provide the RAM coating. The coating may be applied to a substrate to impart the radiation absorbing property thereto.



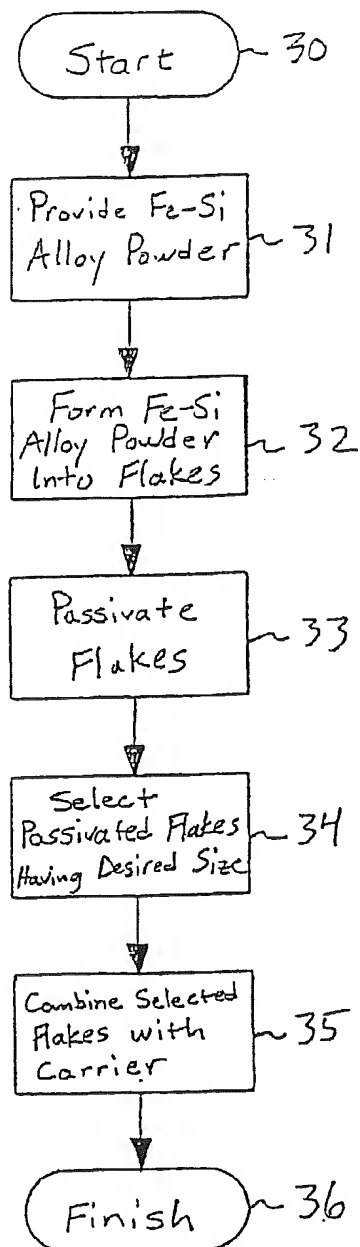


FIG. 3

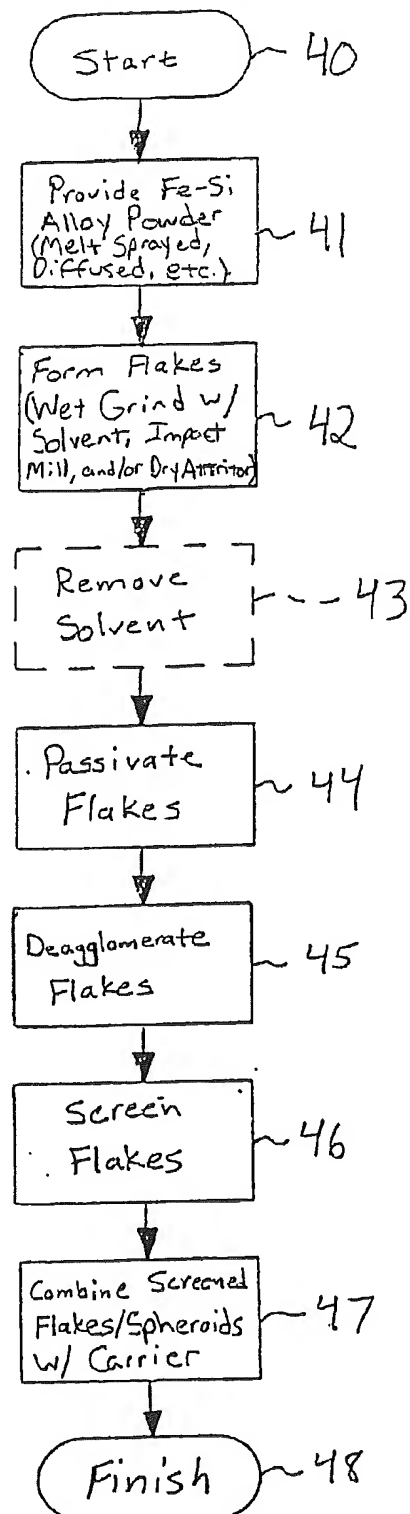
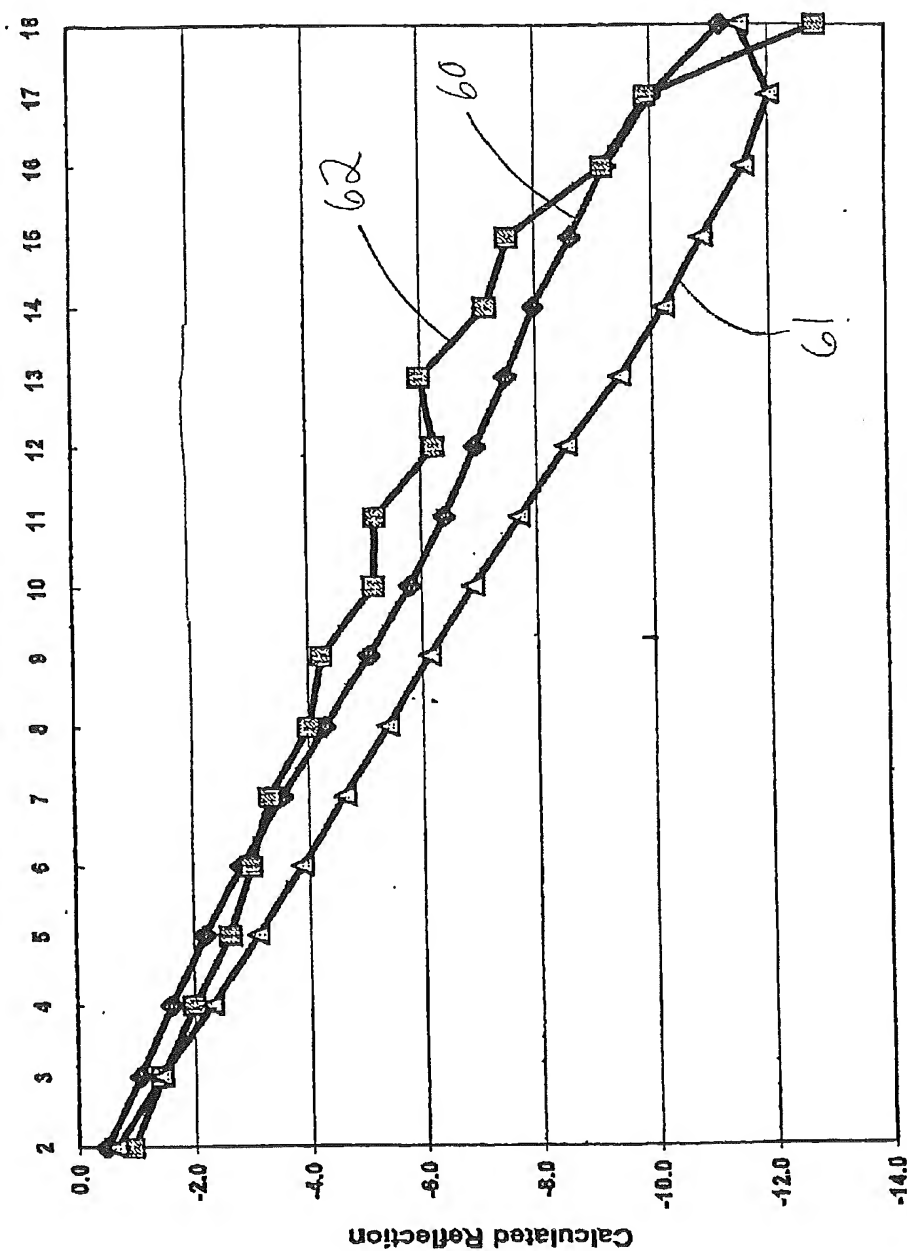


FIG. 4



Frequency (GHz)

FIG. 6

include less than about 25% silicon by weight, as well as less than about 25% Fe_5Si_3 by weight. The passivated iron-silicon alloy flakes may also advantageously include greater than about 40% Fe_3Si by weight and about 0.5-25% FeSi by weight.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] FIG. 1 is a perspective view of a portion of an aircraft having a radiation absorbing material (RAM) coating thereon in accordance with the present invention.

[0014] FIG. 2 is a cross-sectional view of a portion of a wing of the aircraft of FIG. 1.

[0015] FIG. 3 is a flow diagram illustrating a method for making a RAM coating in accordance with the present invention.

[0016] FIG. 4 is flow diagram illustrating the method of FIG. 3 in greater detail.

[0017] FIG. 5 is a graph illustrating in further detail the passivation step of FIG. 3.

[0018] FIG. 6 is a graph illustrating calculated reflection vs. frequency for two RAM materials produced in accordance with the prior art and for a RAM material produced in accordance with the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0019] The present invention will now be described more fully hereinafter with reference to the accompanying drawings, in which preferred embodiments of the invention are shown. This invention may, however, be embodied in many different forms and should not be construed as limited to the embodiments set forth herein. Rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the invention to those skilled in the art. Like numbers refer to like elements throughout.

[0020] Referring initially to FIGS. 1 and 2, a radiation absorbing device in the form of an aircraft 10 in accordance with the present invention is first described. The radiation absorbing device includes a substrate, which in the illustrated example is the airframe 11 of an airplane 10, and a radiation absorbing material (RAM) coating 12 on the substrate. The RAM coating is for absorbing EM radiation incident on the airframe 11, such as radar or other radio frequency (RF) signals, which are illustratively shown by the large arrows 13 in FIG. 1.

[0021] As a result of the RAM coating 12, the amount of EM energy reflected by the airframe 11 will be substantially reduced, as illustrated by the small arrows 14. Thus, the airframe 11 will be more difficult to detect using radar. Of course, those skilled in the art will appreciate that numerous substrates other than airframes (e.g., cables, antennas, etc.) may also advantageously be coated with the RAM coating 12 in accordance with the invention to provide desired EM absorption.

[0022] Turning now additionally to FIG. 3, a method for making the RAM coating 12 in accordance with the invention will now generally be described. The method begins (Block 30) by providing an iron-silicon alloy powder for

processing, at Block 31. Most prior art methods for alloying iron and silicon typically include the compounding of the two powders, adding an activator or catalyst, then sintering the mixture in an electric furnace with an inert atmosphere. By using the catalyst, this reaction becomes exothermic and proceeds quite rapidly at elevated temperatures. This rather violent reaction makes control of the temperature difficult throughout the entire mass of the material

[0023] As a result of the above condition, and coupled with some inhomogeneities in the mixture, this reaction produces more variation in the ferrous silicide phases present than is typically desired. That is, as many as five separate phases may be produced during the alloying process, namely Fe_3Si , $\text{Fe}_{11}\text{Si}_5$, Fe_2Si , Fe_5Si_3 and FeSi . Of these, from the standpoint of developing a RAM material, the α or Fe_3Si is the most desirable. As a result, the alloying reaction is preferably controlled to favor this phase and limit the $\text{Fe}_{11}\text{Si}_5$, Fe_2Si , and Fe_5Si_3 phases. The FeSi phase, which is the equilibrium partner of the Fe_3Si phase, is not as desirable as Fe_3Si in terms of EM absorption, but it also does not have relatively low Curie temperatures as do the $\text{Fe}_{11}\text{Si}_5$, Fe_2Si , and Fe_5Si_3 phases. Accordingly, the FeSi phase is less likely to affect performance at high temperatures, and thus having some FeSi in the starting powder and/or final product is typically not problematic.

[0024] Along with the necessary magnetic characteristics, the RAM powder that is ultimately produced should also have temperature stability and corrosion resistance. The α phase has a very high Curie temperature (greater than 500° C.), as will be appreciated by those of skill in the art, and when the alloy is further processed may develop high corrosion resistance. It will further be appreciated that the effect of composition and temperature may have a significant impact on the phases present.

[0025] Accordingly, it is desirable that the solid state diffusion reaction be carried out such that the iron and silicon alloy in the proper phase ratio to provide the proper starting percentages thereof. Of course, this requires that the reaction temperature be maintained in the necessary range, but this does not always happen as desired due to the rapid and violent reaction rate noted above. As a result, a fused cake, which includes several phases, is often produced using typical prior art approaches. Thus, this fused cake requires reduction to powder and air classification before proceeding with the formation of the corrosion resistant PAM. Yet, the cake is typically very hard and abrasive. Plus, not only is it expensive to reduce the size thereof, but a considerable quantity of undesirable phases may be present in the resulting powder.

[0026] Moreover, in selecting an alloy for use in making RAMs, those of skill in the art will appreciate that it is important to achieve not only reasonably high magnetic moment and high Curie temperature but also to provide high corrosion resistance. The first two characteristics are maximized in the α or Fe_3Si phase. However, this phase is not as resistant to corrosion as are higher percent silicon phases. A balance should thus preferably be achieved in the formulation of the alloy to yield the most optimum properties for reflection or attenuation of radar or other EM radiation, and to perform at temperatures significantly above ambient. That is, the Fe_3Si phase is preferably favored while the $\text{Fe}_{11}\text{Si}_5$, Fe_2Si , Fe_5Si_3 phases are preferably limited, as noted above,

[0039] In particular, as the starting iron-silicon alloy powder preferably includes less than about 25% silicon by weight, the passivated iron-silicon alloy flakes 15 may correspondingly also include less than about 25% silicon by weight. Furthermore, the temperature phases are preferably regulated such that the flakes include less than about 25% Fe_3Si_2 by weight, and rather include greater than about 40% Fe_3Si by weight and about 0.5-25% FeSi by weight.

[0040] Once passivated, the ferrous silicide flakes are removed from the kiln, they are then passed through a de-agglomerator and screened, at Blocks 45 and 46. The former step is desirable as some agglomeration takes place at the 650° C. temperature of the passivation kiln, and the screening allows the passivated flakes 15 having a desired size to be separated from the remainder of the flakes. In particular, the deagglomeration may be performed using a granulator with a 20 mesh barrel screen. Moreover, the flakes may be screened with a screen having openings of about 60 microns or less, for example, to provide the desired flake size and remove any undesirable particles from the refractory containers.

[0041] Stated otherwise, it is typically desirable that the flakes have a maximum dimension 17 (FIG. 2) of less than about 60 microns and, more preferably, about 3 to 20 microns, though other dimensions may also be used. The leftover flakes may then be re-screened, if desired, to increase yield. Of course, in some embodiments screening may be performed to separate flakes of a desired size prior to passivation, but some degree of deagglomeration/screening may still be desirable after passivation depending upon the given application.

[0042] Flakes having such dimensions can then be suspended in a carrier, at Block 47, for later application to the surface of a vehicle, for example, thus ending the method, at Block 48. By way of example, suitable carriers may include organic materials, dielectric materials (e.g., similar to paint, which can be atomized and sprayed on a vehicle), electrically conductive materials, magnetic materials, or a viscous elastomeric material which may be applied in panels. In this latter case the flake size may be made somewhat larger.

[0043] Also, in some embodiments passivated iron-silicon particles having different shapes may be included as well. In particular, in some applications it may be desirable to include not only passivated iron-silicon flakes in the coating but also passivated spherical particles. That is, a base powder with spherical particles may be milled without flaking using one of the above described techniques, passivated, and then deagglomerated and/or screened, as similarly described above. Air classification is an optional step that may be used in conjunction with the milling techniques to provide a desired particle size distribution, as noted above. The passivated spherical particles may then be suspended along with the passivated flakes in various ratios in the carrier to provide different EM absorbing properties (Block 47).

[0044] By way of comparison, plots of calculated reflection vs. frequency are illustratively shown in FIG. 6 for two RAM materials manufactured in accordance with the prior art, and one plot for a RAM material produced in accordance with the present invention including only flaked particles. More particularly, the plot 60 is based upon a ferrous silicide material formed by a prior art diffusion process, the plot 61

is for a material based on carbonyl iron, and the plot 62 is for the RAM made in accordance with the invention. As may be seen, the reflective properties of the material made in accordance with the present invention are generally less than those of the other two materials across most of the illustrated frequency range.

[0045] Many modifications and other embodiments of the invention will come to the mind of one skilled in the art having the benefit of the teachings presented in the foregoing descriptions and the associated drawings. Therefore, it is understood that the invention is not to be limited to the specific embodiments disclosed, and that modifications and embodiments are intended to be included within the scope of the appended claims

That which is claimed is:

1. A method for making a radiation absorbing material (RAM) coating comprising:

- providing an iron-silicon alloy powder;
- forming the iron-silicon alloy powder into flakes;
- passivating the flakes;
- selecting passivated flakes having a desired size; and
- combining the selected passivated flakes with a carrier to provide the RAM coating.
2. The method of claim 1 wherein the iron-silicon alloy powder comprises melt sprayed iron-silicon alloy powder.
3. The method of claim 1 wherein the iron-silicon alloy powder comprises diffused iron-silicon alloy powder.
4. The method of claim 1 wherein forming comprises impact milling the iron-silicon alloy powder.
5. The method of claim 1 wherein forming comprises grinding the iron-silicon alloy powder using a dry attritor.
6. The method of claim 1 wherein forming comprises wet milling the iron-silicon alloy powder in the presence of a solvent.
7. The method of claim 6 wherein the solvent comprises heptane.
8. The method of claim 6 further comprising removing solvent prior to passivating.
9. The method of claim 1 wherein selecting comprises:
 - deagglomerating the passivated alloy flakes; and
 - screening the deagglomerated flakes to obtain flakes having the desired size.
10. The method of claim 1 wherein the desired size is a maximum dimension of less than about 60 microns.
11. The method of claim 1 wherein passivating comprises exposing the flakes to an oxygen containing ambient at a temperature of less than about 700° C.
12. The method of claim 1 wherein passivating comprises passivating the flakes for less than about 24 hours.
13. The method of claim 1 wherein the carrier comprises at least one of an organic material, a dielectric material, an electrically conductive material, a magnetic material, and an elastomeric material.
14. The method of claim 1 wherein the iron-silicon alloy powder comprises less than about 25% silicon by weight.
15. The method of claim 1 wherein combining comprises combining the selected passivated flakes and passivated, generally spherical iron-silicon alloy particles with the carrier to provide the RAM coating.

United States Patent [19]
Kemp, Jr. et al.

[11] **Patent Number:** **4,884,754**
[45] **Date of Patent:** **Dec. 5, 1989**

[54] **PROCESS FOR PRODUCING FINE COPPER FLAKES**

[75] **Inventors:** **Preston B. Kemp, Jr., Troy, N.Y.;**
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[21] **Appl. No.:** **292,788**

[22] **Filed:** **Jan. 3, 1989**

[51] **Int. Cl.⁴** **B02C 19/12**

[52] **U.S. Cl.** **241/5; 241/16;**
241/21; 241/29

[58] **Field of Search** **241/30, 29, 15, 16,**
241/22, 5, 152 A, 24, 21, 172; 75/0.5 R, 0.5 A

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[57] **ABSTRACT**

A process is disclosed for producing fine copper flakes which comprises media milling copper powder particles with one or more organic surfactants in a non-polar organic medium to comminute the copper particles and produce intermediate flakes having a thickness of less than about 3 micrometers, removing the major portion of the organic medium and the organic surfactants from the intermediate flakes to produce dried intermediate flakes, and fluid energy milling the intermediate flakes to reduce the diameter of the dried intermediate flakes and produce flakes having a diameter of no greater than about 10 micrometers in diameter.

5 Claims, No Drawings

At this point the dried intermediate flakes have the desired thickness, but they are generally much too large in flake diameter (largest dimension).

The intermediate flakes are next introduced into a fluid energy mill using high velocity jets of either air or inert gas to entrain the intermediate flakes, impart high velocity to them, and impact them against either a solid substrate or each other. This group of processes is called "jet milling" or "fluid energy milling" including fluidized bed opposed jet milling, the "Coldstream" process in which a stream of gas and the starting material are impinged against a fixed target, etc. All references made herein to "jet milling" or "fluid energy milling" are understood to refer to this group of processes. In the process of the invention, there are no moving parts except for gas compressors to produce the fluid energy stream. Energy is imparted to the particles by the fluid or gas, that is, by the velocity of the fluid. All of these processes impart high velocities to the material which is being ground and impact the accelerated particles against each other or against a solid substrate at a sufficient force to shatter or break the particles into smaller fragments. During this part of the process, the individual flakes tear at cracks present in their outer periphery and are reduced in diameter, while the thickness of the flakes remains the same. In addition, the flakes become more nearly circular when viewed parallel to the thickness direction and the edges are often rounded or rolled up, both which could contribute to less problems in the application as in the screening process in the manufacture of thick films. It is easier to force the rounded edge flakes through the screen during the circuit printing process, than to force a ragged edge flake through. The ragged edges tend to catch on the weave of the screen. Because of the self-classifying nature of typical fluid energy mills, particles above desired upper size limit stay in the milling apparatus until they are reduced in size.

The process of the present invention affords a method for producing copper flake morphology powder at a low cost suitable for use in fabricating thick film electronic circuit boards. One of the primary advantages of the two step process is that the flake thickness and planar dimensions can be independently changed. Powders having the lowest surface area (and therefore, oxygen content) possible to meet the particle size requirements can thus be fabricated.

To more fully illustrate this invention, the following nonlimiting example is presented.

EXAMPLE

Gas atomized copper powder is processed using n-heptane as the milling fluid and oleic acid as the surfactant in a stirred ball mill using tungsten carbide/cobalt media. Proportions of the materials on a weight basis are as follows:

Mill media: 6200 parts
copper powder: 200 parts
n-Heptane: 160 parts
Oleic acid: 1 part.

The powder is milled for about 30 hours at a linear speed of about 364 fpm measured at the outer diameter of the mill agitator to produce flake morphology particles. The particles are removed from the mill as a slurry and filtered and vacuum dried using conventional metal powder processing techniques. The dried particles are next processed in an opposed jet mill using compressed air at inlet pressures of 82-85 psi. The action of the jet mill is to reduce the average diameter of the flake morphology particles without substantially changing their thickness. Size data is shown below:

	Particle Size (Microtrac)		
	Starting powder	Ball milled	Jet Milled
Mean size, microns	14.3	10.0	5.4
Std. deviation, microns	8.4	7.9	4.3

Note that the particle size as measured by laser light scattering is greatly reduced by jet milling. Also, the distribution of particle sizes becomes narrower.

While there has been shown and described what are at present considered the preferred embodiments of the invention, it will be obvious to those skilled in the art that various changes and modifications may be made therein without departing from the scope of the invention as defined by the appended claims.

What is claimed is:

1. A process for producing fine copper flakes, said process comprising:

(a) media milling copper powder particles with one or more organic surfactants in a non-polar organic medium to comminute said copper particles and produce intermediate flakes having a thickness of less than about 3 micrometers;

(b) removing the major portion of said organic medium and said organic surfactants from said intermediate flakes to produce dried intermediate flakes; and

(c) fluid energy milling said intermediate flakes to reduce the diameter of said dried intermediate flakes and produce flakes having a diameter of no greater than about 10 micrometers in diameter.

2. A process of claim 1 wherein said surfactants are fatty acids.

3. A process of claim 2 wherein said surfactants are selected from the group consisting of oleic acid, and stearic acid.

4. A process of claim 1 wherein said non-polar organic medium is n-heptane.

5. A process of claim 1 wherein said media milling step is a stirred media milling step.

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